

CLAIMS

1. A method for producing polyorganosiloxane particles, which comprises the step (A) of hydrolyzing and condensing a silicon compound of the general
5 formula (I),



wherein R^1 , which is a non-hydrolyzable group, is an alkyl group having 1 to 20 carbon atoms, an alkyl group having 1 to 20 carbon atoms and having a
10 (meth)acryloyloxy group or epoxy group, an alkenyl group having 2 to 20 carbon atoms, an aryl group having 6 to 20 carbon atoms or an aralkyl group having 7 to 20 carbon atoms, R^2 is an alkyl group having 1 to 6 carbon atoms and n is an integer of 1 to 3, provided
15 that when a plurality of R^1 s are present, each of R^1 s may be identical to, or different from, other or every other one, and that when a plurality of OR^2 are present, each of OR^2 s may be identical to, or different from, other or every other one,
20 in the presence of a catalyst, to form seed particles of polyorganosiloxane particles and thereby obtaining a seed-particles-containing solution, and the step (B) of mixing said seed-particles-containing solution with a particle-diameter-growing aqueous solution
25 containing a silicon compound of said general formula (I) or a hydrolyzate thereof, to grow said seed particles,

the method comprising the step (A) of obtaining the seed-particles-containing solution in

which, when said silicon compound is dissolved in an aqueous medium, 0.7 to 6.5 mass ppm of a basic catalyst is added to said aqueous medium to cause the silicon compound to undergo preliminary hydrolysis and
5 condensation, and a basic catalyst is added thereto in an amount necessary for forming the seed particles formed of polyorganosiloxane, to form said seed particles.

10 2. The method of claim 1, wherein the basic catalyst is ammonia.

3. The method of claim 1 or 2, wherein the silicon compound of the general formula (I) is
15 methyltrimethoxysilane or vinyltrimethoxysilane.

4. The method of any one of claims 1 to 3, wherein the polyorganosiloxane particles produced have an average particle diameter of over 10 μm and have a
20 particle size distribution whose coefficient of variation (CV value) is 5 % or less.

5. A method for producing polyorganosiloxane particles, which comprises the step (A) of hydrolyzing
25 and condensing a silicon compound of the general formula (I),



wherein R^1 , which is a non-hydrolyzable group, is an alkyl group having 1 to 20 carbon atoms, an

alkyl group having 1 to 20 carbon atoms and having a (meth)acryloyloxy group or epoxy group, an alkenyl group having 2 to 20 carbon atoms, an aryl group having 6 to 20 carbon atoms or an aralkyl group having 7 to 20 carbon atoms, R^2 is an alkyl group having 1 to 6 carbon atoms and n is an integer of 1 to 3, provided that when a plurality of R^1 s are present, each of R^1 s may be identical to, or different from, other or every other one, and that when a plurality of OR^2 are present, each of OR^2 s may be identical to, or different from, other or every other one, in the presence of a catalyst, to form seed particles of polyorganosiloxane particles and thereby obtaining a seed-particles-containing solution, and the step (B) of mixing said seed-particles-containing solution with a particle-diameter-growing aqueous solution containing a silicon compound of said general formula (I) or a hydrolyzate thereof, to grow said seed particles,

the method comprising the step (B) of growing the seed particles, in which the seed particles are measured for diameters continuously or at intervals of a constant time period and the reaction is terminated when an intended particle diameter is reached.

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6. The method of claim 5, wherein the seed particles are measured for diameters continuously or at intervals of a constant time period in a manner in which part of a reaction solution is sampled and

brought into contact with a protective-colloid-forming agent to form a protective colloid on the particles in the reaction solution, and then the measurement is made by a Coalter method.

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7. The method of claim 5 or 6, wherein the polyorganosiloxane particles are polymethylsilsesquioxane particles.

10 8. The method of any one of claims 5 to 7, wherein the particle-diameter-growing aqueous solution containing the silicon compound or a hydrolyzate thereof is added at a rate of 0.01 ml/minute or less per milliliter of volume of the seed-particles-
15 containing solution, for obtaining the polyorganosiloxane particles having a diameter of over 10 μm .

9. The method of any one of claims 5 to 8,
20 wherein the polyorganosiloxane particles finally obtained have a particle diameter of 1 to 30 μm and a coefficient of variation of 3 % or less.

10. A method for producing polyorganosiloxane
25 particles, which comprises the step (A) of hydrolyzing and condensing a silicon compound of the general formula (I),



wherein R^1 , which is a non-hydrolyzable group,

is an alkyl group having 1 to 20 carbon atoms, an alkyl group having 1 to 20 carbon atoms and having a (meth)acryloyloxy group or epoxy group, an alkenyl group having 2 to 20 carbon atoms, an aryl group having 6 to 20 carbon atoms or an aralkyl group having 7 to 20 carbon atoms, R^2 is an alkyl group having 1 to 6 carbon atoms and n is an integer of 1 to 3, provided that when a plurality of R^1 s are present, each of R^1 s may be identical to, or different from, other or every other one, and that when a plurality of OR^2 are present, each of OR^2 s may be identical to, or different from, other or every other one, in the presence of a catalyst, to form seed particles of polyorganosiloxane particles and thereby obtaining a seed-particles-containing solution, and the step (B) of mixing said seed-particles-containing solution with a particle-diameter-growing aqueous solution containing a silicon compound of said general formula (I) or a hydrolyzate thereof, to grow said seed particles,

the method comprising the step (B) of growing the seed particles, in which an anionic surfactant is added to the seed-particles-containing solution to grow the seed particles, the anionic surfactant having a concentration that satisfies the relational expression (II),

$$Y = \alpha \times (a \times X) / (A \times R) \quad \dots (II)$$

wherein a is a theoretical value obtained by dividing a molecular weight of a product that is a

hydrolysis and condensation product of the silicon compound with a molecular weight of the silicon compound,

Y is a concentration (mass %) of the anionic surfactant in the particle-diameter-growing aqueous solution,

X is a mass (g) of the raw material to be used for synthesis of the seed particles,

A is a total mass (g) of the solutions for use in the step (B) of growing seed particles,

R is an average particle diameter (μm) of the seed particles, and

α is a coefficient in the range of $4.0 < \alpha \leq 75$.

11. The method of claim 10, wherein the anionic surfactant has an HLB value of 15 to 40.

12. The method of claim 11, wherein the anionic surfactant is a higher alcohol sulfuric ester salt.

13. The method of claim 12, wherein the higher alcohol sulfuric ester salt is sodium dodecyl sulfate.

14. The method of any one of claims 10 to 13, wherein, in the step (A) of forming seed particles, a separately prepared solution containing polyorganosiloxane particles is added to a reaction system containing the silicon compound, and the silicon compound is caused to undergo hydrolysis and

condensation in the presence of the polyorganosiloxane particles, to form the seed particles.

15. The method of any one of claims 10 to 14,
5 wherein the polyorganosiloxane particles have an average particle diameter of over 10 μm .

16. A method for producing silica particles, which comprises preliminarily calcining the
10 polyorganosiloxane particles obtained by the method recited in any one of claims 1 to 15 at a temperature that is higher than the temperature which is lower than a decomposition temperature of the organic group contained therein by 150°C and that is less than the
15 decomposition temperature of the organic group, and then calcining the polyorganosiloxane particles at the decomposition temperature of the organic group or higher.